August 23, 2013

Via Electronic Mail

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Re: Sampling and Material Retention Plan
Former US Oil Recovery/MCC Recycling Site, Pasadena, Harris County, Texas
CERCLA Docket No. 06-10-11

Dear Ms. Westfall and Ms. Koks:

Per your request, ENVIRON International Corporation (ENVIRON) is pleased to submit this Sampling and Material Retention Plan to the US Oil Recovery Site PRP Group (PRP Group) for collecting and analyzing (for PCBs and dioxins) samples of waste materials at the former US Oil Recovery/MCC Recycling sites (the "Site"). This Sampling and Material Retention Plan provides ENVIRON's proposed approach for sampling and retaining Site waste materials.

Project Understanding

The PRP Group is currently in the process of initiating the removal of waste materials (solids) from the Site and negotiating the scope of work for the Remedial Investigation/Feasibility Study (RI/FS).

The first removal action will involve the removal of oily solids from the Bioreactor, 225 roll-off boxes, one frac tank, and two vacuum boxes at the Site. The PRP Group has asked ENVIRON to prepare an approach for collecting and analyzing samples that are representative of above ground waste contained in the Bioreactor, roll-off boxes, frac tank, and vacuum boxes and to retain samples to preserve the opportunity for performing future evaluation and/or analyses of the material if the need arises.

Based upon a review of the operating history of the former USOR/MCC Recycling facility and historic site documents, there is no reason to expect PCBs to be present in the residual wastes from the USOR operations. Similarly, there is no basis for expecting dioxins to be present above the low background levels commonly associated with the contaminant, now ubiquitous in the environment.

The following factors support this assessment regarding these two classes of compounds:

PCBs

- USOR operations at the Site began in late 2003 when it was permitted as a liquid waste transfer facility. Thus, all operations at USOR occurred long after the strict regulation of PCB-containing materials under the Toxic Substances Control Act (1976). In the intervening years, PCB-containing materials were eliminated from use in most applications and phased out in others. Strict standards on their management and disposal were enacted. PCB-containing oils or other materials could not be legally shipped to the Site.
- Site documents do not suggest the presence of PCBs as a result of USOR operations, and EPA has never considered PCBs as a chemical of concern at the Site.
- PCB analyses performed during EPA's 2010 removal action activities at the Site were all below the method detection limits. The samples were representative and included materials from frac tanks used to collect liquids from roll-off boxes, filter press liquid and sludge phases, liquids from two tanks within the tank farm and liquids in the parking lot.
- Similarly, no PCBs were detected in recent screening-level laboratory analyses performed on 12 roll-off box solids samples collected in preparation for off-site disposal of the solids materials. These materials were from the bioreactor and as such were essentially a composite of materials received at the site over time.

Dioxins

- Dioxins are ubiquitous in the environment and routinely found in low levels as a consequence of lawful combustion processes. As noted by EPA, industrial sources of dioxin to the environment include:
  - Incineration of Municipal Solid Waste
  - Incineration of Medical Waste
  - Coal Fired Power Plants
  - Chlorine Bleaching of Wood Pulp
- None of the above types of operations or their associated industrial processes (chlorination or high temperature combustion) were performed by USOR.
- Historic documents regarding site operations do not suggest the presence of dioxins as a result of USOR operations. EPA has never considered dioxins as a chemical of concern at the Site.

Thus, given the nature of the materials received at the site and the nature and timing of USOR's operations, there is not a basis for including these two classes of contaminants in the list of the chemicals of concern for the USOR operations. To further evaluate this hypothesis in an objective manner and provide further support for removing PCBs and dioxins from the list of analytes in any further study, the PRP Group proposes to sample and analyze certain USOR waste streams prior to their removal from the Site.

Scope of Work

This Sampling and Material Retention Plan is focused on the solids contained in the Bioreactor, roll-off boxes, a frac tanks, and two vacuum boxes located on the USOR property, primarily because the removal of these materials is imminent. The following sections provide an approach for collecting representative samples of Site waste materials and retaining representative materials for potential future testing or evaluation.

Sampling Design

The Bioreactor is an open top concrete-walled structure with two compartments that was previously used as an aeration basin and equalization tank for used oil processing. The compartments, identified as the West Compartment and East Compartment, are equal in size, are separated by an interior concrete wall and appear to be connected via underflow of the interior wall. Historic documentation indicates that the Bioreactor was in operation as an aeration basin and/or equalization tank, and processed Used Oil material.

In order to address the Bioreactor, the PRP Group began the process of removing the liquid and solid contents. Following the removal of accumulated oily storm water, the solids in the Bioreactor were exposed and measured from the top south end of the structure (the only accessible point). The West Compartment contained approximately 3.5 feet of solids across the entire floor and the East Compartment contained approximately 1.5 feet of solids across approximately 60% of the floor. Based on visual observations, the solids appeared to be consistent in both compartments of the Bioreactor and consisted of black oily solids with infrequent areas of reddish-brown hues.

The material in the vast majority of the roll-offs is solids material that was transferred from the Bioreactor over the period of a few days in October 2009. Site documents indicated that 90% of the material in the Bioreactor was transferred to the roll-off boxes. The material is believed to be relatively homogeneous given that it was initially present in the Bioreactor and was transferred to the roll-off boxes over a short period of time. Analytical data for solids samples collected by both the EPA and the PRP group from Bioreactor and some of the roll-offs showed generally similar TCLP analyte concentrations.

Physical observations of material contained in the roll-off boxes following the removal of overlying liquids over the last two weeks also support the homogeneity of the solids within the roll-offs. Of the 225 roll-offs, 212 were observed to contain similar material that closely resembles the material in the Bioreactor (i.e., black oily solids), 4 roll-off boxes were observed to contain brown colored solid material with a more consolidated consistency than the majority of the roll-off boxes, 7 were observed to contain general debris material and plant refuse/trash not derived from the bioreactor, and 2 containers were empty.

Although 4 of the roll-off boxes appear to contain material with a different color and consistency, they will be included as part of the roll-off box sample population for statistical purposes as the wastes are believed to be derived from the same process units and as such should be considered a single waste stream. It should be noted that independent of the random sampling program; two of these four identified roll-off boxes will be sampled in accordance with the protocols described below and in addition to the statistically derived number of randomly selected roll-off boxes. The roll-off boxes that were empty or contained general debris are not included in this Sampling and Material Retention plan.

According to Site documents, material contained in the Bioreactor was transferred into two frac tanks to relief hydraulic stress on the Bioreactor walls following a breach of the top West Compartment concrete wall in 2009. In 2012, one of these two frac tanks was observed to be leaking and as a precautionary measure, the material in the leaking frac tank was transferred to the two vacuum boxes currently on-site.

A sufficient number of the 216 boxes containing solids will be sampled to ensure the material is characterized with acceptable precision and statistical certainty with regard to PCB and dioxin concentrations.

Due to the absence of prior analytical data for dioxin and PCBs, the large number of roll-off boxes and general homogeneity of the materials contained in the Bioreactor and roll-off boxes, a simple random sampling design is the best applicable design for estimating variability and obtaining representative samples from the 216 roll-off boxes and Bioreactor. This design was developed in consideration of potentially applicable USEPA and other guidance, including the following documents:

- USEPA 2002. RCRA Waste Sampling Draft Technical Guidance – Planning, Implementation, and Assessment. EPA 530-D-02-002. http://www.epa.gov/osw/hazard/testmethods/sw846/pdfs/rwsdtg.pdf
- ASTM Standard D6009-12, Standard Guide for Sampling Waste Piles. http://www.astm.org/Standards/D6009.htm
- Isserlis, L. (1918). "On the value of a mean as calculated from a sample". Journal of the Royal Statistical Society (Blackwell Publishing) 81 (1): 75–81.
- USEPA 2006. Data Quality Assessment. Data Quality Methods for Practitioners. EPA QA/G-9S. EPA 240-B-06-003. http://www.epa.gov/QUALITY/qs-docs/g9s-final.pdf
- USEPA. Test Methods for Evaluating Solid Waste, Physical/Chemical Methods (SW846). http://www.epa.gov/osw/hazard/testmethods/sw846/online/#table
- USEPA 2009. Statistical Analysis of Groundwater Monitoring Data at RCRA Facilities. Unified Guidance. EPA530-R-09-007. http://www.epa.gov/osw/hazard/correctiveaction/resources/guidance/sitechar/gwstats/unified-guid.pdf

Sample Size – Roll-off Boxes

The characterization of the chemical composition of the material does not imply or require the characterization of the composition of each individual container (USEPA SW846). USEPA guidance and standard statistical texts describe methods for determining the average chemical composition and the variability associated with that average by sampling a portion of the population. As described in USEPA 2006, 2009 and SW846 the sample size required to characterize the mean with a statistically defined precision is based on the t-statistic. The equation given in EPA 2006, 2009 and SW846 requires selecting the required precession (e.g., +/- 20%), the required statistical power (e.g., 95%), and the standard deviation. However, the equations assume that the samples come from a large population (i.e., >5000). The equation must be adjusted to account for smaller population sizes using the correction for finite populations (Isserlis, 1918):

$$\bar{X} * P = t_{n-1} * \frac{S}{\sqrt{n}} * \sqrt{\frac{N-n}{N-1}} (Eq\ 1)$$

Where t = t statistic

S = estimated standard deviation

n = sample size

N = population size

P = level of precision in the estimate of the mean (e.g., 20%)

As described above, there are a total of 216 roll-off boxes containing the material to be characterized. The relatively small number of boxes means that the equations in EPA 2006, 2009 and SW846 will overestimate the required sample size. For the present evaluation, the sample size required (n) was calculated using equation 1.

The application of equation 1 requires an estimate of the standard deviation. Although screening level testing for PCBs was previously performed on solids from 12 roll-off box samples, no direct quantitative measurements of PCB or dioxin concentrations in the Bioreactor or roll-off box solids are available; however, there are 15 TCLP waste characterization samples that were collected from the 15 roll-off boxes adjacent to the Bioreactor. Benzene was detected in all 15 of the TCLP samples and was selected as a surrogate measure of the expected variability. Consistent with SW846, the mean (2.63) and the standard deviation (1.27) was used to estimate the minimum sample size required to estimate the mean ± 20% using equation 1. The sample size (n) was iteratively varied to determine the minimum sample size require to meet the specified precision and statistical certainty.

Population Size	Precision	Alpha Level	Estimated Sample Size
216	20%	0.05	23
216	20%	0.10	17
216	20%	0.20	11

The sample size analysis for the roll-off boxes shows that a precision of 20% with a 95% certainty (alpha = 0.05) can be attained with a sample size of 23. The results also show that a slightly lower level of statistical certainty (80%, alpha = 0.20 as suggested in SW846 Table 9-1) can be achieved with a sample size of 11.

Based on the sample size calculations, the mean PCB and dioxin concentrations in the roll-off box solids can be measured with a high level of precision and certainty by sampling 23 boxes. Attachment 1 provides the statistical calculations. We recommend collecting and analyzing samples from 25 roll-off boxes to provide an extra level of certainty given the assumptions regarding the expected standard deviation in the PCB and dioxin measurements (SW 846).

Sample Size – Other Containers

ENVIRON proposes to collect separate samples from the Bioreactor, frac tank and vacuum boxes in a manner consistent with the sampling density used for the roll-off boxes. Based on Site history and visual observations, the material in these units is expected to be comparable to that contained in the roll-off boxes. By utilizing a similar sampling density in all wastes, the data set may be combined into a single population for statistical analysis that represents an unbiased random sampling of all wastes, assuming chemical concentrations are found to be consistent. As documented above, 25 samples will be collected from the roll-off boxes. Based on recent solids measurements collected from the roll-off boxes following removal of residual liquid, an estimated total of 1,930 cubic yards of solids are contained in the 216 roll-off boxes to be sampled. Collection of 25 samples from this volume corresponds to a sample density of one sample per 77 cubic yards. The number of samples taken from each non roll-off waste container will be proportional the roll-off box sample density as follows (rounding up to the next integer):

- 5 samples will be collected from the approximate 360 cubic yards in the Bioreactor West Compartment;
- 2 samples will be collected from the approximate 100 cubic yards in the Bioreactor East Compartment;
- 1 sample will be collected from the approximate 50 cubic yards in the frac tank; and
- 1 sample will be collected from the approximate 40 cubic yards contained in the two vacuum boxes (each vacuum box contains approximately 20 cubic yards).

For the waste units described above, the sample will be collected from a random location using the same randomized grid methodology described above for the roll-off boxes. For the vacuum box sample, one grab sample will be collected from one of the two vacuum boxes, which will be randomly selected.

The sample size estimate is based on a random sampling scheme. As noted in the USEPA guidance (EPA, 2002), random sampling should not be interpreted to mean haphazard; rather, it has an explicit statistical meaning of equal probability for selection of a subset from a larger pool. The guidance (specifically Box 3 in Section 5.2.1 on p. 57 of USEPA 2002, and Chapter 9 of SW 846) describes the procedure for randomly selecting grids, time intervals or other units (in this case waste container units such as roll off boxes) for sampling and testing. This procedure provides for dividing a study area into grids or units (i.e., Bioreactor, roll-off boxes, frac tanks and vacuum boxes) for the purposes of randomly selecting sample locations within those units as follows:

- **Roll Off Boxes** – The objective of the roll-off box sampling is to identify specific individual roll-offs to be sampled. For the purposes of this sampling program, each roll off box will be assigned a number between 1 and 216 and a random number generator will be utilized to select the candidate roll off boxes for sampling. The roll off box will then be assigned grids for random selection of a grid cell within the roll-off box for the actual grab sample.
- **Bioreactor** – In the case of the Bioreactor, each compartment of the Bioreactor will be treated independently and assigned independent numbered grids. Selection of sample locations will be performed using a random number generator as with the roll-off box.
- **Frac Tank/Vacuum Boxes** – These containers have limited access to the solids. As such, for the frac tank and two vacuum boxes, the area of the sampling grid will be limited to the area that is safely accessible for sampling and will likely be limited to a small area directly beneath access manways on top of the containers.

The volume of material collected for each sample outlined in the following sections will be at least 5x the minimum sample size requirements dictated by the laboratory for testing. For the purpose of this sampling plan, ENVIRON has assumed each grab sample will be collected in two 8 ounce jars. ENVIRON will consult with the selected laboratory to confirm the appropriate volumes prior to sample collection.

The samples will be submitted to the laboratory for analyses for PCBs and dioxins as detailed below. The laboratory will be requested to save the remaining sample material for a minimum of one year from the date of collection and will be instructed not to discard the samples without prior approval from ENVIRON. Storage costs may be charged to the PRP Group.

Roll-Off Box Sampling

The selected roll-off boxes will be sampled as follows. For each roll-off box randomly selected for sampling, a solids sample will be collected randomly from within the roll-off box by dividing the roll-off into four horizontal quadrants and two vertical layers within the roll-off box such that the roll-off is effectively divided into eight cells. Each cell will be numbered 1 through 8. Using a random number generator, one of the eight cells will be randomly selected for sampling. The grab sample will be obtained using a step ladder located on the outside of the roll-off box without physically entering the roll-off box for health and safety purposes and to avoid disturbing the contents. The sample will be placed in two 8 ounce sample jars by alternately filling each jar with ¼ of the sample volume until each jar is filled.

Bioreactor Sampling

ENVIRON proposes to collect separate samples from each compartment of the Bioreactor since analytical results indicate the solids in the West Compartment are characteristically hazardous and the East Compartment solids are non-hazardous. As stated above, the Bioreactor will be sampled in a manner consistent with the sampling density used for the roll-off boxes. Therefore, five samples will be collected from the West Compartment and two samples will be collected from the East Compartment. The samples will be collected by dividing each compartment into four horizontal quadrants and two vertical layers such that each compartment is effectively divided into eight cells. Each cell will be numbered 1 through 8. Using a random number generator, the appropriate number of cells will be randomly selected for sampling (i.e., five cells for the West Compartment and two cells from the East Compartment. Each sample with be placed in two 8 ounce sample jars by alternately filling each jar with ¼ of the sample volume until each jar is filled.

Frac Tank and Vacuum Box Sampling

Due to the limited access to the solids contained in these containers, samples will be collected directly beneath access manways on top of the containers. One grab sample will be collected from the Frac Tank and one sample will be collected from the two vacuum boxes.

Sampling Procedures

Sampling devices will vary depending on access to the waste and/or container type and will include but may not be limited to hand tools, hand augers, shovels, sludge samplers (e.g., Sludge Judge), Coliwasa Tubes, or mechanical mixers/machinery. In general, samples will be collected using an appropriate sampling device and placed directly in the sample jar.

All sample collection activities will be fully documented, consistent with previous activities at the site. Documentation will include sample log books that document all sample collection information, photographs, copies of chain of custody forms, and copies of shipping labels. The sampling team will immediately contact the project manager if they encounter any unforeseen complications.

Sampling devices and tools will be decontaminated prior to first use on-site and after each sample is collected. The equipment decontamination procedures will consist of the following:

- Non disposable sampling equipment will be scrubbed with a stiff bristle brush using non-phosphate soap and potable water;
- Rinse with potable water; and
- Rinse with distilled water;

Additionally, methanol may be used if necessary to remove oily material prior to final decontamination of the sampling equipment.

Quality Control Procedures

For quality control purposes, field duplicates, equipment blanks and matrix spike/matrix spike duplicates will be collected.

Field Duplicate

Field duplicates will be collected at a frequency of one in every 10 samples collected and will be analyzed for the same suite of parameters as the primary sample. Field duplicates will be labeled and packaged in the same manner as primary samples so that the laboratory cannot distinguish between the primary sample and the duplicate sample.

Equipment Blank

Equipment blanks will be collected at a frequency of one in every 10 samples collected (only when non-dedicated[1] sampling equipment is being used) and will be analyzed for the same suite of parameters as the primary sample to assess the decontamination procedures.

[1] Non-dedicated sampling equipment refers to sampling tools which are designed to be cleaned and re-used for collection of multiple samples.

Matrix Spike and Matrix Spike Duplicate (MS/MSD)

Matrix spike and Matrix Spike Duplicate (MS/MSD) samples will be collected due to anticipated matrix interference (i.e., interference from the oily sample matrix). A minimum of one MS and one MSD will be collected and analyzed for every 20 field samples collected (i.e., collect up to 20 field samples followed by two additional samples designated as MS and MSD).

Analytical Procedures

The samples will be shipped to ALS laboratories in Houston, Texas for PCB analysis by Method 8082A and dioxin analysis by Method 1613B (17 Tetra-Octa dioxin/furan isomers). ALS will utilize method 8082A to measure PCBs in the samples as Aroclors. ALS will subcontract the dioxin analysis to Vista Analytical Laboratory of El Dorado Hills, California. The laboratories will be instructed to ensure that the detection limits for the solid samples are below the most stringent USEPA RSLs for PCBs and dioxins (http://www.epa.gov/region9/superfund/prg/). Due to the oily nature of the samples and the complex analytical matrix, the laboratories will be instructed to utilize enhanced sample cleanup procedures, as allowed by the methods, to minimize the potential impacts of matrix interference.

Validation Procedures

Data validation will be performed by the Project Chemist, who will have experience performing data validation. Consistent with data validation currently underway or proposed for USOR, the data validation steps will be patterned after the USEPA CLP National Functional Guidelines (NFG) for Superfund Organic Methods Data Review and Inorganic Superfund Data Review (USEPA 2008, USEPA 2010). The data validation process will be based on the NFG and analytical methods. The Project Chemist will conduct a Level III validation for all definitive project data and Level IV validation for 10 percent of the data.

Level III data validation techniques include accepting, rejecting, or qualifying the data on the basis of the following QC checks:

- Agreement of analyses conducted with chain-of-custody requests
- Holding times and sample preservation
- Initial and continuing calibrations and analytical sequence
- Instrument tuning (GC/MS only)
- IS performance (GC/MS and ICP-MS only)
- Blanks (laboratory and field QC)
- Surrogate recoveries
- LCS/LCSD results
- MS/MSD and PDS results
- Laboratory duplicate results
- Field duplicate results
- ICP ICS results (AB solution only)
- ICP serial dilution results

Level IV (full) data validation will be performed on 10% of the data and in addition to the above checks will include a review of the raw data (e.g., instrument spectra and chromatograms), backup documentation for calibration standards, analysis run logs, dilution factors, and additional information necessary to check calculations for quantified analytical data. Calculations are checked for QC samples (e.g., MS/MSD and LCS data) and routine field samples (including field duplicates, field blanks, equipment blanks, and VOCs, trip blanks). To verify that detection limits and data values are appropriate, instrument performance, method of calibration and calibration standards are evaluated.

Analytical data may be qualified based on data validation outcomes. Qualifiers will be consistent with the applicable NFG and will be used to provide data users with an estimate of the level of uncertainty associated with the qualified result.

The data validation will be documented in data validation reports that also include the signature of the reviewer and the date of the validation. Data will not be released for use prior to completion of the data validation.

We appreciate the opportunity to assist the USOR PRP Group with this important project. If you have any additional questions or additional comments, please contact me by phone at 713-470-6651 or by email at (bjones@environcorp.com) or Robert Coffman at 405-286-9198 or his email (rcoffman@environcorp.com). We look forward to working with you on this important project.

Sincerely,

Robert E. Coffman, PG
Senior Manager and USOR Project Coordinator

Brent M. Jones, PE, BCEE
Principal and Board Certified Environmental Engineer

Attachment 1 – Calculations

Attachment 1
Calculations

Sample Size Calculation Appendix

$$\bar{X} * P = t_{n-1} * \frac{S}{\sqrt{n}} * \sqrt{\frac{N-n}{N-1}} \ (Eq\ 1)$$

Sample ID	Benzene	Units
1212303-1	1.3	mg/L
1212303-2	3.2	mg/L
1212303-3	2.3	mg/L
1212303-4	3.3	mg/L
1212303-5	3.4	mg/L
1212303-6	2.9	mg/L
1212303-7	3.8	mg/L
1212303-8	4	mg/L
1212303-9	3.3	mg/L
1212303-10	3.3	mg/L
1212303-11	4	mg/L
1212303-12	0.1	mg/L
1212303-13	0.63	mg/L
1212303-14	3	mg/L
1212303-15	0.82	mg/L

Mean	2.623	0.524667
Standard Deviation	1.286	
N	216	

Population Size	Precisison (P)	Alpha	t	n
216	20%	0.05	$t_{(n-1,0.05)}$	23
216	20%	0.1	$t_{(n-1,0.10)}$	17
216	20%	0.2	$t_{(n-1,0.20)}$	11